



Case HF/5-22049/A/PCT

Declaration

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF

Group Art Unit: 1621

FRANK BACHMANN ET AL

SERIAL NO.: 10/031,999

FILED: July 6, 2000

FOR: Metal Complexes of Tripodal Ligands

DECLARATION UNDER RULE 132

I, Dr. Menno Hazenkamp, a citizen of the Netherlands residing in Riehen, Switzerland hereby declare:

1. That I was awarded a PhD degree from the University of Utrecht in Utrecht, The Netherlands;
2. That I have been employed by Ciba Specialty Chemicals as a Chemist since Febr. 1998;
3. That I presently hold the position of a group leader "Stain Removal Development" application research Home and Fabric Care, in Grenzach, Germany;
5. That I consider myself an expert in the field of the application of oxidation catalysts in the field of laundry; and
6. That the application tests of the compounds herein described were performed in my laboratory in strict accordance with my statements in the Declaration.

Ligands which are used in present US application 10/031,999 are disclosed in Japanese Patent Application JP 11-50096. But there is no disclosure that a Mn(III) complex is used. In Examples 1 - 3, the use of a mixture of a pure ligand (LIGAND A; B and C) together with Mn(II)Cl₂ is disclosed.

The following comparison tests have been done to demonstrate that the use of Mn(III) complexes instead of using a ligand and Mn(II)Cl₂ is superior.

The following washing solution has been used for all experiments:

0.6 g base detergent (IEC 60456 A*) per beaker,

8.6 mM H₂O₂ per beaker,

80 ml tap water per beaker and

6 mg/l textile Dye R 81 238 per beaker.

A Linitest apparatus equipped with 8 washing beakers has been used.

The washing time has been 30 min at 40°C.

7.5 g white cotton fabric per beaker (brightness Y = 93) has been used.

The following washing solutions have been prepared:

- I. The washing solution without any complex, ligand and metal.
- II. The washing solution containing 0.3125 g/l of a homogeneous mixture of preformed Mn(III)Saltren complex (0.245%) with Na₂SO₄.

- III. The washing solution containing 0.3125 g/l of a homogeneous mixture containing 0.095% $\text{Mn(II)Cl}_2 \cdot 4\text{H}_2\text{O}$, 3% Saltren ligand and 96.91% Na_2SO_4 . (The molar ratio of Mn and Ligand is equal as in the Japanese Patent Application JP 11-50096, Example 1).

Thus, the solutions II and III always corresponded to a molar concentration of Mn in the liquor of 1.5 μM . After washing, the brightness Y of the white fabric is measured. The results of these test are summarized in the following table

Solution	Brightness Y of cotton fabric after washing
I.	79.6
II.	85.3
III.	80

If there is no active Mn(III)saltren catalyst in the liquor, the fabric has a low brightness Y of 79.6 because the textile dye has caused a blue coloration of the fabric.


If an active Mn(III)saltren catalyst is present in the wash liquor the fabric is brighter, since part of the textile dye has been bleached by the peroxide/catalyst system.

While the preformed Mn(III) complex shows a significant DTI effect (has significantly bleached the textile dye in combination with H_2O_2), the separate addition of $\text{Mn(II)Cl}_2 \cdot 4\text{H}_2\text{O}$ and Saltren ligand shows hardly any significant improvement.

I declare that this behavior could not have been by from a person having ordinary skill in the art.

I, Dr Menno Hazenkamp, finally declare that all statements made herein of my own knowledge are true and that all statements made in information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 25th day of November 2003

A handwritten signature in black ink, appearing to read 'M. Hazenkamp', with a long horizontal line extending to the right.

Menno Hazenkamp